

Comparative Evaluation of the Fluoride Recharge Ability of Two Glass Ionomers Obtained from Fluoridated Dentifrice and CPP-ACFP Paste: An In-vitro Study

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Abstract

Introduction: Glass ionomer cement (GIC) is a restorative material used in pediatric dentistry, which attaches to dental hard tissues and has cariostatic properties due to the ability for fluoride release. The present study aimed to assess the fluoride release and uptake capacity of two GICs in the presence of various fluoride sources. **Methods:** This *in-vitro* study was conducted on 120 disks composed of two GICs (Fuji II LC, Equia Forte System), which were prepared with the exact dimensions of 5×2 millimeters. Fluoride release ability of the samples was determined every 24 hours for seven days and weekly (days 7-21) using a combination of ion selective electrodes. The samples in each group were divided into three subgroups and subjected to no fluoride treatment, fluoridated dentifrice (once a day for one minute), and MI Paste Plus (once a day for one minute). After recharging the samples for seven days, the level of fluoride release was measured on days 1-7, 14, and 21. **Results:** The results of one-way analysis of variance indicated that the fluoride release ability of Fuji II was higher compared to that of EQUIA Forte ($P<0.001$). In addition, fluoridated dentifrice could recharge both the glass ionomers more significantly than the MI Paste Plus. **Conclusion:** According to the results, light-cured, resin-reinforced glass ionomers could release significantly higher levels of fluoride compared to EQUIA Forte. Moreover, the fluoride rerelease was higher by the GICs when recharged with fluoridated dentifrice compared to the MI Paste Plus.

Keywords: Casein Phosphopeptide-Amorphous Calcium Fluoride Phosphate, Dentifrices, Fluoride, Glass Ionomer.

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Introduction

Dental caries is a prevalent chronic disease, which particularly affects young children. Dental professionals have been concerned with the restoration of carious teeth with minimal aggression to the tooth structure using cariostatic materials (1, 2). Glass ionomer cements (GICs), also known as resin infiltration, are used as a minimally invasive approach involving the removal of tissue decay using manual instruments alone and resin sealants (3). In this technique, restoration of carious teeth with fluoride-releasing restorative materials has been proposed as a possible mechanism to reduce the occurrence of secondary caries. In addition, these materials diminish the counts of residual bacteria under the restoration (4, 5).

GICs are essential materials for such purposes due to their releasing of fluoride and chemical adhesion to the tooth structure (6). Glass ionomers are able to take up and rerelease fluoride ions from exogenous sources (7, 8). Fluoridated dentifrices are considered to be the most common sources of fluoride with the capability of daily use. In this regard, Freedman et al. (9) had denoted that home care fluoride exposure provides sufficient measurable fluoride uptake and rerelease. Furthermore, Rao has stated that fluoride is diffused into the GIC matrix material and increases its fluoride reservoir, from which it is gradually released (10). However, several factors may affect the process of fluoride release from GICs, such as formulation, solubility, and porosity of the material (11).

Recently, a new restorative material has been introduced (EQUIA Forte, GC, Tokyo, Japan), which contains a high-viscosity, conventional GIC (EQUIA Fil, formerly known as Fuji IX GP extra), as well as a novel nanofilled coating material (EQUIA Coat, formerly known as G-coat plus). According to the indications of the manufacturer, EQUIA Forte is optimal for class I, II, and V restorations. The self-adhesive, nanofilled resin (G-coat) of EQUIA Forte infiltrates the surface of GICs, thereby providing long-lasting protection and marginal integrity and increasing the strength and wear resistance of the GIC surface (12, 13).

According to the literature, casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) has anticariogenic properties (14, 15). The combination of CPP-ACP and fluoride (CPP-ACFP) has been reported to enhance the incorporation of fluoride into the plaque and subsurface enamel, which could substantially improve the remineralization of the subsurface lesions in the enamel (16).

To the best of our knowledge, no prior studies have evaluated the use of CPP-ACFP paste to recharge GICs.

The tested null hypotheses of the current research were as follows:

- 1) There is no difference between the fluoride-releasing properties of the two selected glass ionomers;
- 2) There is a difference between fluoride rerelease with exposure to various fluoride sources.

The present study aimed to investigate the fluoride release and uptake capacity of two glass ionomers in the presence of various fluoride sources.

Materials and Methods

This *in-vitro* study was conducted using light-cured, resin-reinforced GIC (group I, Fuji II, GC Corporation, Japan) and bulk fill hybrid GIC (group II, EQUIA Forte System, GC, Japan) (Table I). The capsules of the materials were only activated before mixing, set into the amalgamator (ultram2, SDI, Australia), and triturated for 10 seconds. In total, 60 specimens were prepared in each group and placed in customized Teflon molds (diameters: 5×2 mm). During the fabrication of the specimens, the top and bottom surfaces of the molds were covered by polyester strip, supported by glass slabs on either side, and clamped in order to provide a smooth surface. The excess extruded material was removed by gentle pressure.

In accordance with the instructions of the manufacturer, the setting time of EQUIA Forte GIC is 2.5 minutes since the beginning of mixing. The EQUIA coat was applied and photocured for 20 seconds using dental curing light (Coltolux 75, Coltene Whaledent, Switzerland). The light-cured, resin-reinforced GICs from each surface were polymerized for 20 seconds.

GIC disks were removed from the molds and stored in a humid environment at the temperature of 37°C for 24 hours. Afterwards, all the specimens were suspended independently in plastic bottles containing five milliliters of deionized water and placed in an incubator at the constant temperature of 37°C. After 24 hours since the preparation of the suspension, the first fluoride concentration was measured. The deionized water in the plastic container was buffered with total ionic strength adjustment buffers (TISAB II) in order for stable pH, as well as to prevent the generation of fluoride ion complexes with various cations. In addition, five milliliters of the storage media was mixed with five milliliters of TISAB II, and the fluoride level was assessed using a digital ion analyzer and fluoride electrode (Mettler Toledo, United States). The instrument was calibrated with a series of standard fluoride solutions at the concentrations of 0.50, 1.00, 2.00, 10.00, 20.00, and 100 ppm through diluting the fluoride standard of 1,000 mg/l. The specimens were

transferred to a new bottle, and the solution was refreshed every 24 hours for the first week, followed by weekly refreshment for 21 days (17, 18).

After measuring the fluoride release for 21 days, the samples were divided into three subgroups. Each subgroup of specimens was subjected to one treatment, including no fluoride treatment (subgroup A; control), application of fluoridated dentifrice (subgroup B; Oral B 1450 ppm; once a day for one minute), and application of MI Paste Plus (subgroup C; CPP-ACFP 900 ppm; once a day for one minute) (Table II).

After the treatment, each disk was wiped clean with a tissue and placed in five milliliters of deionized water

for 24 hours. The treatments were repeated during the first week, and fluoride measurement was performed every 24 hours. In the second week, no treatment was carried out, and the fluoride release of the samples was measured every 24 hours on days 7-14 and 21 (8, 19).

Data analysis was performed in SPSS version 17 using Shapiro-Wilk test to assess the normal distribution of the data. In addition, repeated measures analysis of variance (ANOVA) and Tukey's HSD post-hoc test were applied ($\alpha=0.05$), and the P-value of less than 0.05 was considered statistically significant.

Table I. Description of Materials Used in Study

Material	Manufacturer	Composition
Oral B	Oral B Laboratories, London, UK	Hydrated silica, sodium hexametaphosphate, PEG-6, propylene glycol, aqua zinc lactate, sodium gluconate, CI 77891, sodium lauryl sulfate, silica, aroma, sodium saccharin, chondrus crispus powder, trisodium phosphate, stannous fluoride, stannous chloride, xanthan gum, and sodium fluoride (1,450 ppm)
MI Paste Plus	GC America, Alsip, Illinois, USA	Pure water, glycerol, CPP-ACP, D-sorbitol, CMC-Na, propylene glycol, silicon dioxide, titanium dioxide, xylitol, phosphoric acid, sodium fluoride, flavoring, sodium saccharin, ethyl p-hydroxybenzoate, propyl p-hydroxybenzoate, and butyl P-hydroxybenzoate.

Table II. Examined Pastes in Study

GIC Type	Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 14	Day 21
Fiji II	1.6726±0.034	0.4058±0.025	0.3658±0.048	0.2640±0.027	0.25440±0.038	0.2750±0.036	0.2354±0.041	3.6852±0.035	2.4948±0.03
EQUA	1.8414±0.034	0.4904±0.02	0.1860±0.033	0.1138±0.025	0.10260±0.03	0.1574±0.019	0.0832±0.032	0.8714±0.02	0.6846±0.024

Results

Mean values of fluoride release (ppm) in the samples of the glass ionomers are presented in Table III. Accordingly, the mean values showed a significant reduction in the fluoride release from day one until day seven in both groups ($P<0.001$). On days 14 and 21, cumulative fluoride release was observed. According to our findings, there were statistically significant differences between various days of the experiment in this regard ($P<0.001$). Moreover, the results of one-way ANOVA indicated that fluoride release was higher in Fuji II compared to EQUIA Forte ($P<0.001$). Until day seven, the mean values of fluoride release (daily recharge in subgroup B) were significantly higher compared to subgroups A and C ($P<0.001$). However,

no significant difference was denoted between subgroups A and C in this regard ($P=0.135$) (tables IV&V).

After seven days of daily recharge, no significant difference was observed in terms of fluoride release between subgroups A, B, and C of group two on days 1-7, 14, and 21 of specimen incubation ($P=0.416$). Furthermore, the fluoride release following the recharge between subgroups A, B, and C of group one was showed a significant difference from day one until day 21 ($P<0.001$). Significant differences were also denoted in the mean values between groups one and two ($P<0.001$) (Tables VI & VII).

Table III. Mean value of fluoride release for two types of GIC

Material	Manufacturer	Composition
Equia Fil	GC, Tokyo, Japan	Powder: 95% strontium fluoroalumino-silicate glass, 5% polyacrylic acid Liquid: 40% aqueous polyacrylic acid
Equia Coat	GC, Tokyo, Japan	50% Methyl methacrylate, 0.09% camphorquinone Liquid Polyacrylic acid (20–22%) 2-Hydroxyethyl methacrylate (HEMA) (35–40%) Proprietary ingredient (5–15%)
Fiji II	GC, Tokyo, Japan	2,2,4, Trimethyl hexamethylene dicarbonate (5–7%) Triethylene glycol dimethacrylate (4–6%) Powder Alumino-fluoro-silicate glass (100%) (Powder/liquid ratio: 0.33/0.10 g)

Table IV. Mean value of fluoride uptake of light-cured resin-reinforced GIC (Fiji II) after seven days of daily recharge

	Day1	Day2	Day3	Day4	Day5	Day6	Day7
No treatment	.2198±.013	.0684±.036	.0572±.021	.05±.026	.0508±.034	.045±.032	.484±.033
Oral B	1.1488±.025	.78120±.027	.54680±.035	.511±.031	.4532±.037	.41880±.025	.35060±.043
MI paste plus	.3708±.019	.2092±.019	1214±.033	.1374±.029	.27580±.038	.122±.035	.1046±.018

Table V. Mean fluoride uptake of Equia Forte after seven days of daily recharge

	Day1	Day2	Day3	Day4	Day5	Day6	Day7
No treatment	.0634±.0027	.03132±.0031	.025±.0025	.0236±.0035	.0196±.0027	.018±.0015	.0174±.0016
Oral B	1.6692±.051	.5664±.06	.41046±.027	.3005±.014	.3164±.03	.2806±.036	.2362±.031
MI paste plus	.1498±.035	.0878±.054	.0656±.027	.0434±.027	.04±.02	.04±.036	.0468±.034

Table VI. Mean value fluoride release of light-cured resin-reinforced GIC(Fuji II) following recharge

	Day1	Day2	Day3	Day4	Day5	Day6	Day7	Day14	Day21
No treatment	.0432±.037	.0508±.037	.0428±.021	.0428±.01	.0414±.01	.0604±.032	.0406±.034	1.2858±.205	1.871±.167
Oral B	.1364±.033	.154±.067	.1008±.022	.2004±.029	.0776±.061	.0636±.071	.05±.012	1.9478±.195	2.1864±.146
MI paste plus	.0534±.036	.2528±.027	.0566±.0019	.0442±.004	.0558±.003	.0554±.0016	.052±.0012	1.7044±.195	2.2362±.195

Table VII. Mean value fluoride release of Equia Forte following recharge

	Day1	Day2	Day3	Day4	Day5	Day6	Day7	Day14	Day21
No treatment	.1646±.027	.015±.007	.0124±.001	.013±.0017	.0146±.005	.0146±.001	.014±.001	.5092±.03	.7066±.024
Oral B	.0952±.002	.032±.002	.0184±.001	.01±.001	.0134±.005	.0158±.008	.0126±.001	1.4494±.037	1.5536±.038
MI paste plus	.2752±.034	.0852±.001	.0576±.004	.0576±.007	.0382±.002	.0458±.001	.0378±.008	.6004±.019	1.0948±.014

Discussion

Among various fluoride-releasing restorative materials, GICs have the widest application. The main advantages of GICs include the ease of handling, adhesion to enamel and dentin, and biocompatibility. The fluoride release and recharge ability of GIC plays a pivotal role in the prevention of recurrent caries and remineralization of incipient carious lesions (20, 21). Moreover, GICs reduce the bacterial count under the restoration through fluoride release (21).

In the present study, we also evaluated the initial fluoride release from two different glass ionomers during 21 days, as well as the fluoride rerelease after recharging with two fluoridated pasts. According to the obtained results, the fluoride release by Fuji II GICs was higher compared to that of EQUIA Forte GICs. We utilized two GICs, including light-cured, resin-reinforced GIC and EQUIA Forte GIC, in the form of pre-dosed capsules in order to avoid errors in mixing and prevent the improper calibration of the proportions between the powder and liquid. These materials were applied after trituration using an amalgamator. The finishing and polishing of the specimens could change the surface area of the materials, and the specimens with no surface treatment were

investigated. EQUIA Forte GIC was a combination of a packable glass ionomer and a self-adhesive, nanofilled coating, wherein the resin coating could optimize its physical properties. In the clinical evaluation of EQUIA GIC at 12-, 24-, and 36-month intervals, the properties were found to be similar to those of resin composite (22).

Some fluoridated materials (e.g., mouth rinses, pastes, and dentifrices) could be used for the fluoride count recharge of GICs. Therefore, the current research was designed to evaluate the effects of Oral B fluoride dentifrice and MI Paste Plus on the fluoride recharge ability of GICs. Several studies have investigated the efficacy of CPP-ACFP in preventing demineralization and promoting the remineralization of early enamel lesions (13, 23). For instance, Liena et al. (15) observed that within a period of four weeks, CPP-ACFP was superior to fluoride varnish in terms of remineralizing smooth-surface white spot lesions, while CPP-ACP exerted no such effect.

The *in-vitro* fluoride release from GICs could be influenced by various factors, including the fluoride concentration in the materials, size and composition of the inorganic filler, powder-liquid ratio of two-phase systems, mixing procedure, curing time, inner-material

porosity, surface treatment, exposed area of the specimen, and type, temperature, and pH of the utilized immersion media (24, 25). In the present study, deionized water was used as the storage medium. Deionized water is considered to be an absolute means to the assessment of fluoride release from restorative materials since it contains no fluoride traces (26).

Fluoride ion-selective electrode was applied in order to analyze the fluoride concentration released by the study groups in the current research. According to the findings, maximum fluoride released by the glass ionomers occurred within the first 24 hours and decreased during first week. This phenomenon could be explained based on the initial burst hypothesis, which is caused by the reaction of glass particles to polyalkenoic acid during the setting reaction (26). These findings are in line with the previous studies in this regard, which have demonstrated similar fluoride release patterns (8, 24-28). This consistency could be attributed to the initial fluoride release from the surface as low levels of fluoride continued to be released during the following days owing to the ability of fluoride to diffuse through cement pores and fractures (8, 27).

According to the results of the present study, fluoride release was significantly higher in the light-cured, resin-reinforced glass ionomer compared to EQUIA Forte during the first week. The higher fluoride release by the light-cured, resin-reinforced GICs could be due to the slowed acid-based reactions by the resin component compared to conventional GICs. This slow reaction makes the ionic matrix less mature and capable of releasing more fluoride, thereby increasing the porosity of resin-reinforced GICs (28, 29). Previous findings have indicated that resin-modified GICs exhibit higher fluoride release and uptake capacity in the long run compared to conventional GICs (26). Correspondingly, Cabral MFC et al. (27) have reported wide variations in the amounts of the fluoride ions released in restorative materials, which could not be attributed to the category of cement as conventional or resin-modified GICs.

EQUIA Forte is a glass hybrid material, which represents the most recent innovation in glass ionomers and resin technologies with EQUIA Forte Fil and EQUIA Forte Coat in synergy. In a study in this regard, Hattab et al. (30) stated that the surface coating agent interfered with microleakage, significantly reducing the ionomer cement fluoride release in deionized water and artificial saliva.

After day 21 of fluoride release in the current research, two concentrations of fluoride were used for the recharge of the specimens for seven days. After exposure to fluoridated materials, fluoride release increased in the

two GIC groups. Consistent with the previous studies in this regard, our findings indicated that exposure to fluoridated dentifrices or MI Paste Plus allowed the material to take up fluoride (31). In addition, the quantity of fluoride release was significantly higher in subgroup B (Oral B) compared to subgroups A (control) and C (MI Paste Plus). The difference between the values obtained in subgroup B and other subgroups might be due to the lower level of fluoride in MI Paste Plus (CPP-ACFP 900 ppm) compared to fluoridated dentifrices (1,450 ppm). While fluoride release was higher in subgroup C compared to subgroup A, no significant difference was observed between these subgroups in terms of fluoride release. This phenomenon could be due to the fact that the ability of GIC to reuptake fluoride from preventive materials may occur at high concentrations of fluoride.

In another research, Poggio et al. (32) investigated the fluoride release and uptake ability of various fissure sealants after exposure to fluoridated varnish (5% sodium fluoride) and paste (MI Paste Plus). According to the obtained results, fluoride varnish recharged the sealants significantly more than highly fluoridated toothpaste.

In the present study, fluoride recharge significantly decreased in the GIC specimens with fluoride exposure for one week during days 1-7. Furthermore, fluoride release was significantly lower in the GIC specimens that were exposed to fluoride for one week compared to the first 21 days regardless of fluoride treatment. After recharging, the most rapid release in both materials occurred on the first day, followed by a significantly lower yet continuous fluoride release after 21 days. This finding is in congruence with the previous studies, which used fluoridated dentifrice to improve the recharge ability (19, 21).

According to the current research, the mean value of fluoride release 21 days after daily recharge was significantly higher in the resin-reinforced glass ionomer compared to EQUIA Forte. In general, the materials with higher initial fluoride release have higher recharge ability comparatively (33).

One of the limitations of this *in-vitro* study was difficulty in simulating the oral environment. Fluoride release was measured in the specimens immersed in a static medium, and the dynamic nature of the condition of the oral cavity might have been overlooked. Therefore, it is recommended that further investigations be performed so as to confirm these findings.

Conclusion

According to the results, the resin-reinforced glass ionomer released significantly higher levels of fluoride compared to EQUIA Forte. In addition, the fluoridated

dentifrices (Oral B Laboratories, UK) could recharge both the glass ionomers more significantly compared to CPP-ACFP paste (MI Paste Plus).

Conflicts of Interest

None declared.

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